

Effect of Sintering on the Physical and Mechanical Properties of Co-Cr-Mo (F-75)/HAP Composites

(Kesan Persinteran ke Atas Sifat Fizikal dan Mekanikal Komposit
Co-Cr-Mo (F-75)/HAP)

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ABSTRACT

This paper reports on the effects of HAP addition and sintering temperature on the microstructure and properties of the F-75/HAP composites fabricated by powder metallurgy. Co-Cr-Mo (F-75) is used in orthopedics because of its excellent biocompatibility when implanted to human or animal body. Hydroxyapatite (HAP) powders have been used as fillers because HAP is the one of the most effective biocompatible materials with similarities to mineral constituents of bones and teeth. HAP powders (chemical formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) have been added to Co-Cr-Mo alloys in composition of 0 to 10 wt. %. The mixtures were then milled, cold compacted at 550 MPa, before sintered at 1100 and 1200°C in a tube furnace. The density, porosity, microhardness and compressive strength were measured. The composites that have been sintered at temperature 1200°C showed better physical and mechanical properties than those produced at 1100°C. After sintering at 1200°C, the samples show higher density, compared with the sample sintered at 1100°C. The sample with no HAP which have been sintered at 1200°C has the highest microhardness (208.9 HV), compared with the same sample sintered at 1100°C (194.3HV). As the temperature is increased from 1100 to 1200°C, the value of compressive strength increased from 184.538 to 341.086 MPa. Microstructural analysis for line scan showed that, as the sintering temperature was increased, there was good interface bonding between HAP particles and matrix F-75.

Keywords: Co-Cr-Mo alloys; hydroxyapatite; mechanical properties; physical properties; sintering

ABSTRAK

Kertas ini melaporkan tentang kesan penambahan HAP dan suhu persinteran ke atas mikrostruktur dan sifat komposit F-75/HAP yang difabrikasi menggunakan kaedah metalurgi serbuk. Co-Cr-Mo (F-75) terkenal digunakan dalam bidang ortopedik kerana keserasian bio yang amat baik apabila digunakan di dalam badan manusia atau haiwan. Serbuk hidroksiapatit (HAP) telah digunakan sebagai bahan penambah kerana HAP adalah salah satu bahan yang mempunyai keserasian bio yang amat efektif bersama dengan kandungan mineral untuk tulang dan gigi. Serbuk HAP (formula kimia $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) telah ditambah kepada aloi Co-Cr-Mo dalam komposisi 0 sehingga 10 peratus berat. Campuran ini kemudiannya diadun, ditekan sejuk pada 550 MPa, sebelum disinter pada suhu 1100 dan 1200°C di dalam relau tiub. Ketumpatan, keliangan kekerasan mikro dan kekuatan mampatan telah diubah. Komposit yang telah disinter pada suhu 1200°C menunjukkan sifat fizikal dan mekanikal yang lebih baik berbanding komposit yang disinter pada suhu 1100°C. Selepas disinter pada suhu 1200°C, sampel menunjukkan nilai ketumpatan yang tinggi, jika dibandingkan dengan sampel yang disinter pada suhu 1100°C. Untuk keputusan ujian kekerasan, sampel tanpa HAP yang telah disinter pada suhu 1200°C mempunyai nilai kekerasan mikro yang paling tinggi (208.9 HV), jika dibandingkan dengan sampel yang sama yang telah disinter pada suhu 1100°C (194.3 HV). Untuk ujian kekuatan mampatan, apabila suhu bertambah daripada 1100 ke 1200°C, nilai kekuatan mampatan bertambah daripada 184.538 ke 341.086 MPa. Analisis mikrostruktur untuk imbasan garisan menunjukkan bahawa, apabila suhu persinteran bertambah, wujudnya ikatan antaramuka yang baik antara partikel HAP dan matrik F-75.

Kata kunci: Aloii Co-Cr-Mo; hidroksiapatit; persinteran; sifat fizikal; sifat mekanikal

INTRODUCTION

In general, biomaterials are used to make devices to replace a part or a function of the body in a safe, reliable, economic and physiologically acceptable manner (Hench & Erthridge 1982). The development and use of biomaterials are expected to continuously increase over the coming decades as a result of ageing populations in Europe, China, Japan

and the USA (Narayan 2010). Variety of materials including polymers, metals, ceramics and composites with the appropriate physical properties and biocompatibility are chosen for the fabrication of biomaterials. Cobalt based alloys become one of the important metallic biomaterials because they exhibited low levels of corrosion and this alloy remains a fixture in orthopedic surgery to this day.

A Co-Cr based alloy which is well known for its high Young's modulus, fatigue strength, wear resistance, good biotolerance and corrosion resistance is an important metallic biomaterial. In particular, the second very important group of materials used in implantology is ceramic, with much attention has already been paid to calcium based biomaterials like hydroxyapatite (HAP with chemical formula $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), due to their excellent biocompatibility, structural and chemical similarity with bone mineral compositions (Shekhar et al. 2010). As reported by Navarro et al. (2008), the application of this ceramic material as bone substitutes started around the 1970s and has mainly used as bone defect fillers.

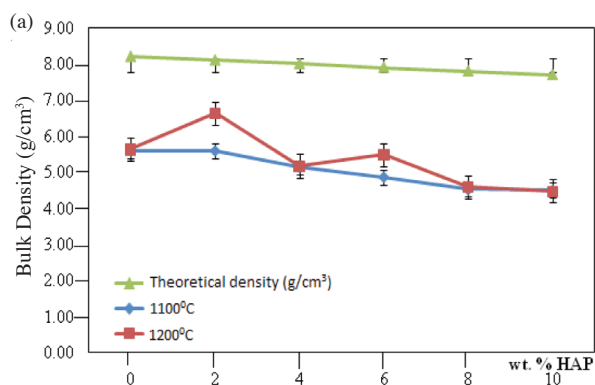
Very limited researches have been reported about bio-composite material based on Co-Cr-Mo matrix filled with HAP, but the results showed that Co-Cr-Mo/HAP is a promising candidate for biomaterial implants to replace hard tissue from the point of view of physical and mechanical properties (Shamsul et al. 2007). Therefore, the objective of this research was to study the effect of HAP addition and sintering temperature in Co-Cr-Mo/HAP composite produced by powder metallurgy method.

MATERIALS AND METHODS

The investigations were carried out on Co-Cr-Mo powders with the addition of hydroxyapatite (HAP) varied from 0 to 10 wt. % of HAP. The 3 wt. % stearic acid was added as a binder. Table 1 shows the partition of the composites in wt. %.

TABLE 1. The partition of composites in wt. %

Sample	HAP [wt. %]	Co-Cr-Mo (F-75) [wt. %]
F-75/0HAP	0	100
F-75/2HAP	2	98
F-75/4HAP	4	96
F-75/6HAP	6	94
F-75/8HAP	8	92
F-75/10HAP	10	90



The samples have been milling in rotary milling machine for 20 min at 154 RPM. The cylindrical green compacts of 13 mm in diameter and 14 mm in height were cold compacted under the pressure of 550 MPa (7.44 tonne). The samples were then sintered at two different sintering temperatures which were 1100 and 1200°C for 2 h in a tube furnace under argon atmosphere. The heating rate was set at 3°C/min until it reaches 400°C and soaking for 30 min. The heating rate was then increased to 5°C/min until the sintering temperature reaches 1100°C and 1200°C and soaking for 2 h before cooling down to the room temperature.

All the sintered densities and porosity were estimated by Archimedes principle method. Vickers microhardness and compression tests were carried out for all sintered samples. Microstructural observation was carried out using scanning electron microscope (SEM) (model JEOL, JSM-6420LA). The sample for this observation was prepared by the standard metallographic methods. A line scan technique was used to analyze the microconstituents in the microstructure (Shi et al. 2010) and the bonding between HAP and F-75.

RESULTS AND DISCUSSION

The influence of sintering temperature on the density and porosity of the fabricated samples is presented in the plots of Figure 1(a) and 1(b). Figure 1(a) shows the plot for theoretical density and bulk density for samples sintered at 1100 and 1200°C. The theoretical density of

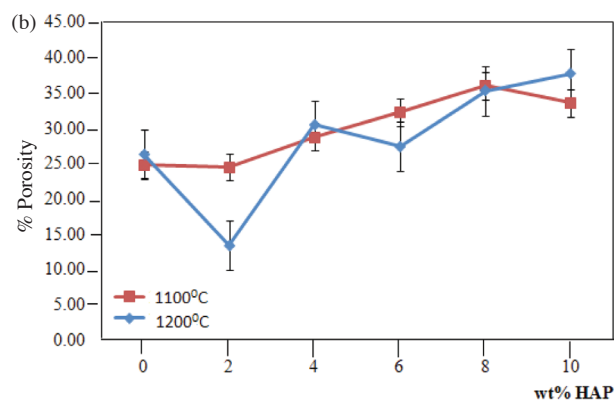


FIGURE 1. The results of (a) bulk density and (b) % of porosity for samples sintered at 1100°C and 1200°C, with different addition of HAP

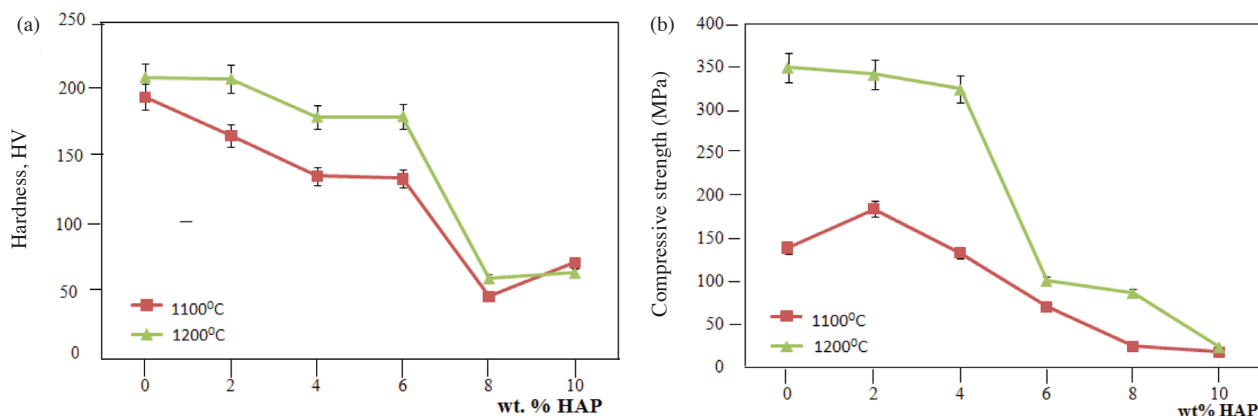


FIGURE 2. The results of (a) hardness and (b) compressive strength for samples sintered at 1100 and 1200°C, with different addition of HAP

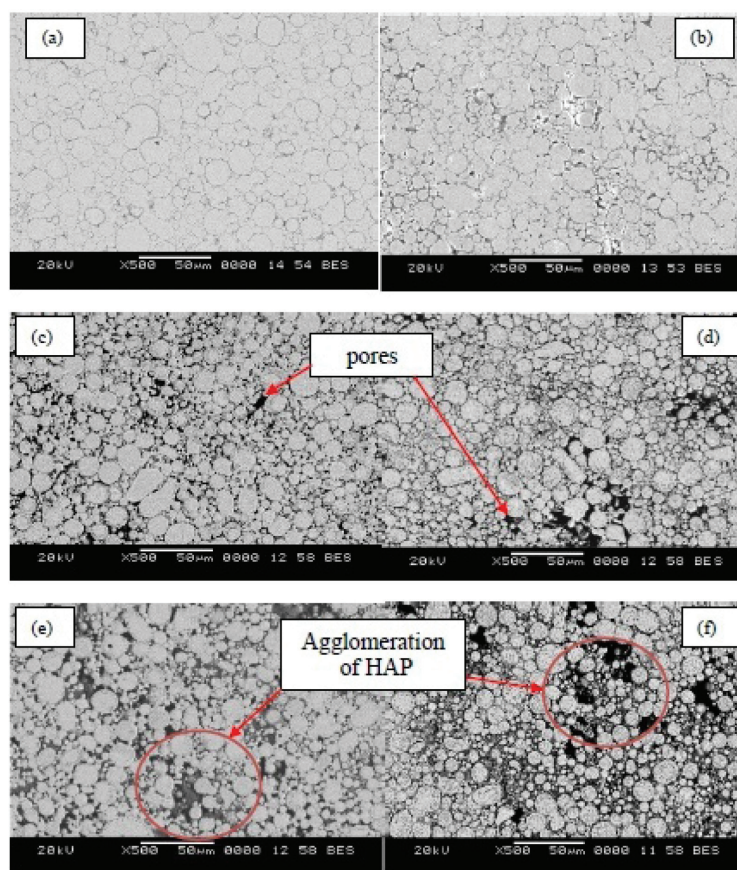


FIGURE 3. SEM micrographs for samples (a) with no HAP, (b) with 2 wt. % HAP, (c) with 4 wt. % HAP, (d) with 6 wt. % HAP, (e) with 8 wt. % HAP and (f) with 10 wt. % HAP after sintered at 1100°C

the composites was calculated from the following formula (Oksiuta et al. 2009): $X\% \times \text{theoretical density of the Co-Cr-Mo alloy} + Y\% \times \text{theoretical density of HAP}$. The theoretical density of the Co-Cr-Mo alloy is 8.2 g cm^{-3} , whereas the theoretical density of HAP is 3.16 g cm^{-3} (Park & Lakes 2007).

Figure 1(a) shows that the trend of theoretical density and bulk density is the same, which is due to addition of HAP, density value was decreased. From Figure 1(a), after

sintering at temperature 1200°C, the samples show higher density compared with the sample sintered at 1100°C, with the highest density comes from sample with 2 wt. % of HAP (6.62 g cm^{-3}). This indicates that during sintering, diffusion reaction between the Co-Cr-Mo powder and HAP particles takes place (Oksiuta et al. 2009).

Pores are necessary for tissue formation, because they allow migration and proliferation of cells, as well as vascularization (Mour et al. 2010). Figure 1(b) which

presents the plot for percent of porosity calculated from the value of density in Figure 1(a). According to the plots in Figure 1(b), F-75/2HAP which has been sintered at 1200°C showed the lowest percentage of porosity (13.31%). This porosity result is related to the result of compression in Figure 2(b), which confirms that the decrease in compressive strength is due to increasing of porosity in HAP ceramics (Le Huec et al. 1995).

Figure 2(a) shows the plot for hardness values while Figure 2(b) is the result of compressive strength for the composites after sintered at 1100 and 1200°C. Both results showed the same pattern, which is as more addition of HAP to F-75 alloys, the values of hardness and compressive strength will decrease. For hardness results, based on the trend, the reference sample (without HAP) at temperature 1200°C has the highest microhardness value (208.9 HV), compared with reference sample at temperature 1100°C (194.3 HV). Compressive strength results also showed the same trend, as the sintering temperature is increased from 1100 to 1200°C, the strength is also increased. The sample with 2 wt. % of HAP shows the highest value which is 184.538 MPa for 1100°C and increased to 341.086 MPa for 1200°C at the same addition of HAP. Both results indicated that the strength and hardness of F-75/HAP alloy is directly proportionate with sintering temperature (Ghazali et al. 2010).

Figures 3 and 4 show the microstructures of the F-75/HAP composite after sintering at 1100 and 1200°C. The SEM micrograph shows that more addition of HAP created more pores in the microstructure, so the value of microhardness and compressive strength (in Figure 2(a) and 2(b)) tend to be lower. The percentage of porosity (Figure 1(b)) has become higher as more HAP is added which proved in microstructure of the composites in Figures 3(b)-3(f) and 4(b)-4(f). As we can see in Figures 3 and 4, more agglomeration of HAP associated with the pores as more HAP is added (Figures 3(b)-3(f) and 4(b)-4(f)). According to Dourandish (2008), the microstructure of parts made from the coarse powder consists of relatively homogenous large pores can be suitable for biomedical application.

SEM-EDS analysis indicates (Figure 5) that two main metallic powder elements (Co and Cr, presented in light blue and purple line colour) react with Ca from HAP (in yellow line) and diffuse towards the amorphous structure of reinforcing phase (HAP). Therefore, it can be summarized that bonding between two composites constituents exists that influences the density and properties of the specimens (Oksiuta et al. 2009). From Figure 5, we can see that sintering at higher temperature (1200°C) (Figure 5(b)) shows better bonding, which the reaction takes place almost nearly high to the line scan.

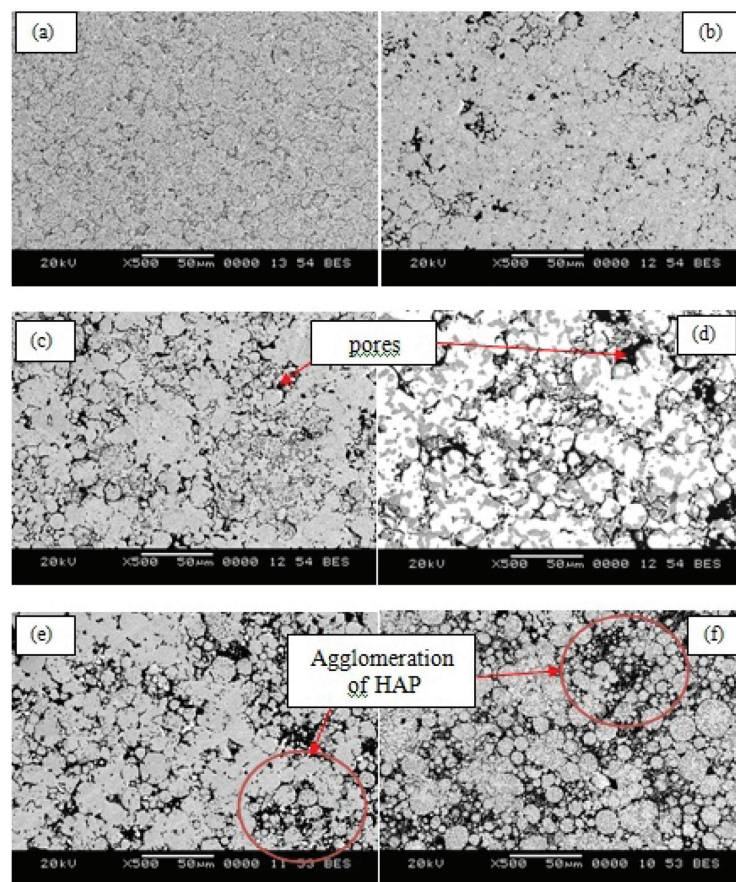


FIGURE 4. SEM micrograph for sample (a) with no HAP, (b) with 2 wt. % HAP, (c) with 4 wt. % HAP, (d) with 6 wt. % HAP, (e) with 8 wt. % HAP and (f) with 10 wt. % HAP after sintered at 1200°C

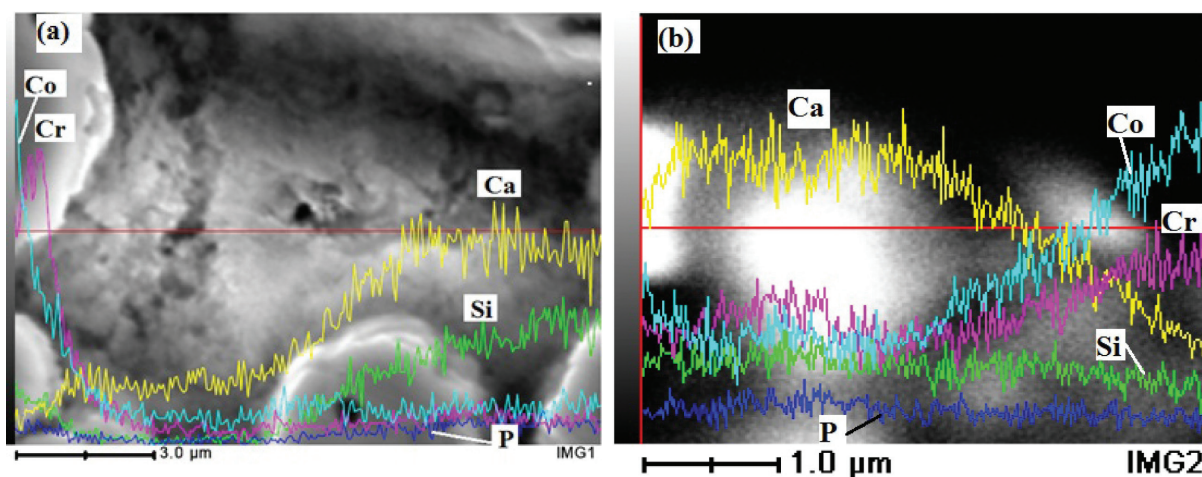


FIGURE 5. SEM- EDS Line scan of sample F-75/10HAP after sintered at (a) 1100°C and (b) 1200°C

CONCLUSION

In this paper, we have reviewed the effect of sintering temperature on the physical and mechanical properties of F-75/HAP composites. The F-75/HAP composites in the range of HAP addition between 0-2 wt. % of HAP which have been sintered at temperature 1200°C showed better physical and mechanical properties than those produced at 1100°C. The microstructural observation shows that more addition of HAP can create more pores. Good interface bonding between HAP particles and F-75 was observed for the sample sintered at 1200°C.

ACKNOWLEDGEMENTS

The authors would like to thank the Higher Education Ministry Malaysia for the financial support via FRGS Grant (9003-00212) and Sustainable Engineering Cluster Grant, under Universiti Malaysia Perlis (UNIMAP).

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Received: 7 March 2012

Accepted: 31 May 2012